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INTELLECTUAL PROPERTY LAW

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Sir:

Transmitted herewith for filing is the patent application of

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For: POWDER COATING

Enclosed are:

- ☒ A specification consisting of 122 pages  
 \_\_\_ sheet(s) of \_\_\_ drawings  
☒ An assignment of the invention  
 \_\_\_ Certified copy of Priority Document(s)  
☒ Executed Declaration ☒ Original \_\_\_ Photocopy  
 \_\_\_ A verified statement to establish small entity status under 37  
 CFR 1.9 and 37 CFR 1.27  
 \_\_\_ Preliminary Amendment  
 \_\_\_ Information Disclosure Statement, PTO-1449 and reference(s)  
 \_\_\_ Other \_\_\_\_\_

20250 U.S. PTO

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Respectfully submitted,

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## POWDER COATING

## BACKGROUND OF THE INVENTION

Field of the Invention

5           The present invention relates to a combination of two  
or more powder coatings usable in a powder coating method  
for forming a coating film having visually homogeneous hue  
by mixing two or more powder coatings of which each color  
is different, and to a coating method for forming a  
10       coating film having visually homogeneous hue using the  
above powder coatings.

Discussion of the Related Art

15           Conventionally, powder coatings have been prepared by  
adding numerous color pigments to resins, curing agents,  
additives, blending the above components, melt-kneading  
the mixture, followed by cooling, pulverization, and  
classification. Also, powder coatings prepared by  
dry-blending two or more primary colored powder coatings  
20       having an average particle size of 10  $\mu\text{m}$  or less for color  
adjustment, and granulating the obtained powders to a  
particle size of from 30 to 50  $\mu\text{m}$  have been reported, as  
disclosed in Japanese Patent Laid-Open No. 7-188586, of  
which the disclosure is incorporated herein by reference.  
25       However, in these powder coatings, the powder coatings

have to be prepared for each color needed, so that an enormous number of powder coating products are needed. Similarly, in order to simplify the toning process, as disclosed in Japanese Patent Unexamined Publication No.

5 4-504431, which corresponds to USP 5,319,001, of which the disclosure is incorporated herein by reference, there has been proposed to provide a powder coating prepared by forming a composite of several kinds of colored powders, each of colored powders, having a particle size of  
10 preferably 10  $\mu\text{m}$  or less, the composite being granules comprising agglomerates of colored powders and having a particle size of from 15 to 75  $\mu\text{m}$ , whereby the different colors applied during coating cannot be discerned by the human eyes.

15 However, even if these colored powders were very fine, having a particle size of 10  $\mu\text{m}$  or less, it is actually not easy to form a coating film of visually homogeneous hue depending upon the combination of two or more colored powders. Moreover, those powder coatings  
20 need a granulation process or a composite-forming process, thereby making it difficult to prepare powder coatings of desired hue in a simple process.

Also, when a powder coating is used by homogeneously mixing two or more powder coatings having different  
25 colors, there are some cases where white pigments are used

for giving good covering ability. However, when white pigments are contained in all of the powder coatings blended, the resulting coating film tends to be inferior in chroma.

5           Accordingly, an object of the present invention is to provide a combination of powder coatings usable in a powder coating method for forming a coating film having visually homogeneous hue by mixing two or more powder coatings of which each color is different.

10           Another object is to provide a powder coating composition usable in a powder coating method for forming a coating film having visually homogeneous hue, the powder coating composition comprising two or more powder coatings.

15           A still another object is to provide a coating method using the powder coating of the present invention.

          These and other objects of the present invention will be apparent from the following description.

20                               SUMMARY OF THE INVENTION

          As a result of intense research in view of the above problems in the prior art, the present inventors have found that differences in the properties of, for instance, pigments usable in powder coatings, cause unevenness in  
25   the triboelectric chargeability of each powder, which in

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turn leads to color unevenness, thereby making it difficult to form coating films having visually homogeneous hue. Therefore, the present inventors have found that a coating film having visually homogeneous hue is formed by controlling the differences in the triboelectric charges of each powder within a given range. The present invention has been completed based upon these findings.

More specifically, the present invention is concerned with the following:

(1) A combination of powder coatings usable in a powder coating method for forming a coating film having visually homogeneous hue by mixing two or more powder coatings of which each color is different, wherein each of differences in triboelectric charges of two or more powder coatings is 5.0  $\mu\text{C/g}$  or less;

(2) The combination of powder coatings described in item (1), wherein each of differences in true specific gravities of two or more powder coatings is 0.15 g/cc or less;

(3) The combination of powder coatings described in item (1) or item (2), wherein each of differences in apparent densities of two or more powder coatings is 0.020 g/cc or less;

(4) The combination of powder coatings described in any

one of items (1) to (3), wherein each of differences in softening points of two or more powder coatings is 5.0°C or less, the softening points being measured using a capillary rheometer;

5 (5) The combination of powder coatings described in any one of items (1) to (4), wherein each of differences in dielectric constants of two or more powder coatings is 0.20 or less;

10 (6) The combination of powder coatings described in any one of items (1) to (5), wherein each of ratios of the electric resistance of two or more powder coatings is from 0.1 to 10;

15 (7) The combination of powder coatings described in any one of items (1) to (6), wherein at least one powder coating is a white powder coating containing a white pigment, and the remaining powder coatings comprise no white pigments;

20 (8) A powder coating composition usable in a powder coating method for forming a coating film having visually homogeneous hue, wherein the powder coating composition comprises two or more powder coatings selected from the combination of any one of items (1) to (7);

25 (9) A coating method for forming a coating film having visually homogeneous hue, comprising the step of applying onto a substrate to be coated two or more powder coatings

of which each color is different, wherein each of differences in triboelectric charges of two or more powder coatings is 5.0  $\mu\text{C/g}$  or less;

(10) The coating method described in item (9), wherein  
5 each of differences in true specific gravities of two or more powder coatings is 0.15 g/cc or less;

(11) The coating method described in item (9) or item (10), wherein each of differences in apparent densities of two or more powder coatings is 0.020 g/cc or less;

10 (12) The coating method described in any one of items (9) to (11), wherein each of differences in softening points of two or more powder coatings is 5.0°C or less, the softening points being measured using a capillary rheometer;

15 (13) The coating method described in any one of items (9) to (12), wherein each of differences in dielectric constants of two or more powder coatings is 0.20 or less;

(14) The coating method described in any one of items (9) to (13), wherein each of ratios of the electric resistance  
20 of two or more powder coatings is from 0.1 to 10;

(15) The coating method described in any one of items (9) to (14), wherein at least one powder coating is a white powder coating containing a white pigment, and the remaining powder coatings comprise no white pigments;

25 (16) A coating method for forming a coating film having



visually homogeneous hue, comprising the steps of applying a white coating onto a substrate to be coated to give a substrate coated with the white coating; and then applying thereon two or more powder coatings of which each color is different, wherein each of differences in triboelectric charges of two or more powder coatings is 5.0  $\mu\text{C/g}$  or less;

(17) The coating method described in item (16), wherein each of differences in true specific gravities of two or more powder coatings is 0.15 g/cc or less;

(18) The coating method described in item (16) or item (17), wherein each of differences in apparent densities of two or more powder coatings is 0.020 g/cc or less;

(19) The coating method described in any one of items (16) to (18), wherein each of differences in softening points of two or more powder coatings is 5.0°C or less, the softening points being measured using a capillary rheometer;

(20) The coating method described in any one of items (16) to (19), wherein each of differences in dielectric constants of two or more powder coatings is 0.20 or less; and

(21) The coating method described in any one of items (16) to (20), wherein each of ratios of the electric resistance of two or more powder coatings is from 0.1 to 10.

# DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a combination of powder coatings usable in a powder coating method for forming a coating film having visually homogeneous hue by mixing two or more powder coatings of which each color is different, whereby a coating film having visually homogeneous hue is obtained by applying onto a substrate to be coated two or more powder coatings. In the present specification, the coating film having "visually homogeneous" hue means that the hue of the formed coating film is homogeneous to such an extent that the colors of individual powder coatings blended therein cannot be discerned by human eyes.

The powder coating of the present invention comprises a resin, a curing agent, an additive, and a colorant.

The resins usable in the present invention may be any of conventionally known resins in this field without particular limitation. Examples of the resins include non-reactive resins, such as polyethylenes, nylon resins, and vinyl chloride; and reactive binder resins, such combinations as epoxy resins/amines, epoxy resins/acid anhydrides, polyester resins/melamine resins, self-curing acrylic resins, polyester resins/epoxy resins, and acrylic resins/polybasic acid resins. For instance, in the present invention, suitable examples of the binder resins

include polyester resins, epoxy resins, and acrylic resins.

The curing agents usable in the present invention may be any of conventionally known curing agents without particular limitation. The curing agents are preferably one or more members selected from polyisocyanate compounds, isocyanurate compounds, blocked isocyanate compounds, epoxy compounds, alkoxysilane compounds, polyethyleneimines, and oxazoline compounds.

Examples of the polyisocyanate compounds include tolylene diisocyanate, and xylylene diisocyanate. Examples of the isocyanurate compounds include 1,3,5-triglycidyl isocyanurate (hereinafter simply referred to as "TGIC"). Examples of the blocked isocyanate compounds include those prepared by blocking the reactive isocyanate group of the above polyisocyanate compounds or their prepolymers with known blocking agents, such as lactam compounds and oxime compounds. Examples of the epoxy compounds include bisphenol A diglycidyl ethers. Examples of the alkoxysilane compounds include methoxysilane oligomers and ethoxysilane oligomers. Examples of the polyethyleneimines include adipic acid dihydrazide and succinic acid dihydrazide. Examples of the oxazoline compounds include 1,4-bis(2-oxazolinyl-2)benzene and

1,2,4-tris(2-oxazolinyl-2)benzene. The amounts of these curing agents may be an amount effective for crosslinking, which may be dependent on the amounts of the functional groups present in the binder resin. More preferably, the  
5 contents of the curing agents are added in an amount of from 0.8 to 1.2 equivalent of the functional groups.

The additives usable in the present invention may be any of known ones without particular limitations. Examples thereof include levelling agents, such as  
10 acrylate polymers, crosslinking accelerators, such as various catalysts and organotin compounds, pinhole preventives, such as benzoin. Each of the additives may be preferably added in an amount of from about 0.1 to about 5 parts by weight, based on 100 parts by weight of  
15 the resins.

Regarding the colorants, the powder coating of the present invention can be formulated with or without a white pigment. Examples of the white pigments include titanium oxide, calcium carbonate, barium sulfate, and  
20 zinc flower, with a preference given to titanium oxide from the aspect of covering ability. The amount of the white pigments is preferably from 5 to 60 parts by weight, based on 100 parts by weight of the resin. Also, any other conventionally known colorants may be used without  
25 particular limitations, which may be suitably selected

depending upon the desired tone. Examples of those colorants (preferably for powder coatings comprising no white pigments) include carbon blacks, copper phthalocyanine, acetoacetic acid arylamide-based monoazo yellow pigments, and pigment reds. The amount thereof is preferably from about 5 to about 60 parts by weight, based on 100 parts by weight of the resin.

The powder coating of the present invention may be prepared by melt-kneading each of the above components using an extruder. After cooling the resulting kneaded mixture, it is subjected to physical pulverization using pulverizing devices, such as hammer mills and jet mills, and then the pulverized product is classified using classifiers, such as air classifiers and micron-classifiers, to give powder coatings of desired average particle sizes. The powder coating of the present invention preferably is a powder having an average particle size of from 1 to 50  $\mu\text{m}$ , more preferably from 5 to 30  $\mu\text{m}$ . Further, from the viewpoint of easy handling, the average particle size is more preferably from 10 to 20  $\mu\text{m}$ . The average particle size of the powder is preferably 50  $\mu\text{m}$  or less, from the aspect of having a sufficiently thin coating film, and the average particle size is preferably 1  $\mu\text{m}$  or more, from the aspect of preventing the agglomeration of the particles, thereby

making it possible to achieve good homogeneous blending.  
In the powder coating of the present invention, a visually  
homogeneous coating film can be easily prepared even when  
the particle size of the coating to be applied has a size  
5 exceeding 10  $\mu\text{m}$ , as in the case of having a size of 10 to  
20  $\mu\text{m}$  as mentioned above.

Also, in the present invention, flowability  
controlling agents, such as silica, alumina, titania, and  
zirconia may be further added to the surface of the powder  
10 coating.

Next, the combination of the powder coatings of the  
present invention will be explained in detail below.

(1) In the present invention, the triboelectric charges  
of the powder coatings are controlled within a desired  
15 range. Methods for controlling triboelectric charges  
include a method by adjusting acid values, amine values,  
etc. of the resin; a method by adjusting charges and  
amounts of the colorants; a method by adding various  
additives, such as quaternary ammonium salts, dyes, and  
20 metallic soaps; and a method by adjusting the amounts of  
such agents as silica, alumina, titania, and zirconia, the  
agents being added to adjust flowability. For instance,  
in a case where a resin having a high acid value is used  
as a raw material, negative triboelectric charges  
25 increase.

In the present invention, an example of a method for measuring triboelectric charges of each of powder coatings is as follows. First, a coating powder is applied using the coating machine and the substrate to be coated under working conditions but without curing. After air blowing-off of the powder from the substrate, a difference in voltage owing to the efflux of electric charges from the substrate and the amount of the powder coatings removed are measured. Alternatively, as a simple method, the triboelectric charge is measured by a blow-off method when each of the powder coatings is blended with various powder coatings having particle sizes larger than that of the powder coating to be tested.

In the present invention, each of differences in triboelectric charges of two or more powder coatings is 5.0  $\mu\text{C/g}$  or less. In other words, in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the triboelectric charges between A and B, between B and C, and between A and C is 5.0  $\mu\text{C/g}$  or less. When the difference in the triboelectric charges exceeds 5.0  $\mu\text{C/g}$ , the electric lines of forces are likely to be concentrated during coating at the edge portion of the substrate to be coated, so that those having high triboelectric charges are likely to be agglomerated at the edge portion, thereby making it

difficult to obtain a coating film having visually homogeneous hue.

(2) In the present invention, the dielectric constant of the powder coating may be further controlled as a property relevant to the triboelectric charges. Methods for controlling dielectric constants include a method by adjusting dispersed particle sizes of various additives in a binder resin; a method by adjusting dielectric constants of workable resins, colorants, etc., wherein the dielectric constant increases when a binder resin or a colorant having a high dielectric constant is used; a method by adjusting a used amount of the colorants, with a preference given to a case where colorants having different dielectric constants are used in combination in order not to change its hue; a method by adjusting the amount of barium titanate, a high dielectric; a method by adding various additives, such as quaternary ammonium salts, dyes, and metallic soaps; and a method by adjusting the amounts of such agents as silica, alumina, titania, and zirconia, the agents being added to adjust flowability. As needed, these methods may be used singly or in combination so as to better control the dielectric constant.

In the present invention, the dielectric constant of each of powder coatings may be calculated by obtaining a



capacitance (Cp) which is measured using an LCR meter "HP16451B" (manufactured by Yokogawa-Hewlett-Packard Ltd.).

In the present invention, each of differences in dielectric constants of two or more powder coatings is preferably 0.20 or less, particularly 0.10 or less. In other words, it is preferred that in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the dielectric constants between A and B, between B and C, and between A and C is 0.20 or less. The difference in the dielectric constants is preferably 0.20 or less, from the aspect of giving an even coating for each color, thereby making it possible to obtain a coating film having visually homogeneous hue.

(3) In the present invention, the electric resistance of the powder coating may be further controlled as a property relevant to the triboelectric charges. Methods for controlling electric resistance include a method by adjusting kinds and amounts of the above colorants and resins; and a method by adding a conductive compound, such as conductive titanium oxide "EC-300" (manufactured by Titan Kogyo K.K.) in the powder coating or to the surface of the powder coating. Here, a preference is given to a case where the electric resistance is controlled by adding

a colorless conductive compound to the powder coating from the aspect of giving desired hue in the powder coating.

In the present invention, the electric resistance of each of powder coatings may be calculated by obtaining a  
5 conductance (G) which is measured using an LCR meter "HP16451B" (manufactured by Yokogawa-Hewlett-Packard Ltd.).

In the present invention, each of differences in electric resistance, as calculated under the above  
10 conditions, of two or more powder coatings is preferably from 0.1 to 10, particularly from 0.5 to 2. In other words, it is preferred that in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the electric resistance between  
15 A and B, between B and C, and between A and C is from 0.1 to 10. The difference in the electric resistance is preferably 0.1 to 10, from the aspect of giving an even coating for each color, thereby making it possible to obtain a coating film having visually homogeneous hue.

(4) In the present invention, from the aspect of blending the powder coatings in a homogeneous mixture and applying the mixture, it is preferred to use a combination of powder coatings with substantially the same level of true specific gravity. In the present invention, methods for  
25 controlling true specific gravities include a method by

adding such additives as titanium oxide, calcium carbonate, barium sulfate, and zinc oxide to the powder coating. Here, from the aspect of giving the desired hue of the powder coatings, a preference is given to a case where zinc oxide is added, as zinc oxide does not affect the hue. The amount of the additives may be suitably chosen so as to adjust the difference in the true specific gravities between each of the powder coatings within the range specified in the present invention. The amount of the additives is preferably from 5 to 60 parts by weight, more preferably from 5 to 40 parts by weight, based on 100 parts by weight of the resin.

In the present invention, the true specific gravity of a powder coating may be, for instance, measured by a conventional method using Micromeritics Multivolume Pycnometer 1305 (manufactured by Shimadzu Corporation).

In the present invention, each of differences in true specific gravities of two or more powder coatings is preferably 0.15 g/cc or less, particularly 0.10 or less. In other words, it is preferred that in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the true specific gravities between A and B, between B and C, and between A and C is preferably 0.15 g/cc or less. The difference in the true specific gravities is preferably 0.15 g/cc or

less, from the aspect of preventing the separation to each  
of powder coatings owing to the difference in the true  
specific gravities by, for instance, the vibration of the  
blended two or more powder coatings during transportation,  
5 so that differences in the blending ratios of the powder  
coatings during coating for each application are less  
likely to take place. As a result, the difference between  
the hue of initial coating and the hue after coating many  
coatings can be maintained to be small. In other words,  
10 not only the hue of each coating film is visually  
homogeneous, but also the hue between the coating films is  
also visually homogeneous.

(5) In the present invention, from the aspect of blending  
the powder coating in a homogeneous mixture and applying  
15 the mixture, it is preferred to use a combination of  
powder coatings with substantially the same level of  
flowability. Methods for controlling flowability include  
a method by controlling average particle size, particle  
size distribution, and particle forms of the powder; a  
20 method by adding agents for adjusting flowability, such as  
silica, to the powder surface. These methods may be used  
in combination. The average particle size and the  
particle size distribution of the powder may be controlled  
using a classifier. Also, the particle forms may be  
25 controlled by adjusting the retention time in the

pulverizing machine. The powder may be spheroidized by a hot air treatment after pulverization. The methods for adding agents for adjusting flowability, such as silica, to the powder surface including a method using a

5 high-speed agitator, such as a Henschel mixer and a Super Mixer. In the case where the agents for adjusting flowability, such as silica, are added to the powder coatings, the amount of the agents used may be suitably selected so as to adjust the apparent density of each of  
10 the powder coatings within the range specified in the present invention. The amount of the agents is preferably from 0.01 to 5 parts by weight, more preferably from 0.05 to 2 parts by weight, based on 100 parts by weight of the powder coating.

15 In the present invention, the apparent density of each of powder coatings may be measured by a conventional method using a powder tester (manufactured by Hosokawa Micron Corp.). In the present invention, each of differences in apparent densities of two or more powder  
20 coatings is preferably 0.020 g/cc or less. In other words, it is preferred that in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the apparent densities between A and B, between B and C, and between A and C is  
25 preferably 0.020 g/cc or less. The difference in the

apparent densities is preferably 0.020 g/cc from the aspect of preventing the agglomeration of each color upon blending the powder coating, so that differences in the blending ratios of the powder coatings during coating for each application are less likely to take place. As a result, not only the hue of each coating film is visually homogeneous, but also the hue between the coating films is also visually homogeneous.

(6) In the present invention, from the aspect of uniformly stoving upon applying the powder coating, it is preferred to use a combination of powder coatings with substantially the same level of melting properties.

Methods for controlling melting properties may be any one of methods by adjusting the melting properties of the resin generally employed. In other words, the adjustment may be made by selecting a suitable molecular weight, molecular weight distribution and monomers constituting the resins for the workable resins. Further, in order to finely controlling the melting properties, the amount of the additives may be adjusted. In the present invention, each of the differences in the softening points in the combination of two or more powder coatings is preferably 5.0°C or less.

In the present invention, the softening point of each of the powder coatings may be measured using a capillary

rheometer "SHIMADZU CFT-500 FLOW TESTER" (manufactured by Shimadzu Corporation), the equipment conventionally used in measurement of softening points for various resins.

5 In the present invention, each of differences in softening points of two or more powder coatings is preferably 5.0°C or less. In other words, it is preferred that in a case, for example, where three kinds of powder coatings A, B, and C are blended, each of the differences in the softening points between A and B, between B and C, 10 and between A and C is preferably 5.0°C or less. The difference in the softening points is preferably 5.0°C from the aspect of giving similar melting state upon stoving of the each of the powder coatings, thereby giving a coating film having visually homogeneous hue.

15 The present invention is concerned with a combination of two or more powder coatings of which each color is different, wherein various properties, including triboelectric charges, are controlled by means mentioned above. A preference is given to a combination where at 20 least one powder coating is a white powder coating containing a white pigment and the remaining powder coatings comprise no white pigments. In conventional methods, when a white pigment is contained in all of the powder coatings, only those with blurred hue and low 25 chroma can be obtained. By contrast, in the method of the

present invention, a wide variety of hue can be obtained depending upon the proportions of the each powder coatings added, from those having high lightness and chroma and those having low lightness and chroma.

5           In the present invention, two or more powder coatings may be blended by any one of conventionally known blending methods, including, for instance, dry-blending methods using high-speed mixers, such as a Henschel mixer and a Super Mixer. The amount of each powder coating may be  
10           suitably selected depending upon the desired hue by mixing various colors.

          The present invention also provides a powder coating composition usable in a powder coating method for forming a coating film having visually homogeneous hue, wherein  
15           the powder coating composition comprises a combination of two or more powder coatings mentioned above.

          The powder coating composition of the present invention may be prepared by the method for blending two or more powder coatings mentioned above.

20           The powder coating of the present invention may be usable in a powder coating method for forming a coating film having visually homogeneous hue, comprising the step of applying onto a substrate to be coated two or more powder coatings of which each color is different. In  
25           other words, the coating method of the present invention



is to form a coating film having visually homogeneous hue, comprising the step of applying onto a substrate to be coated two or more powder coatings of which each color is different, wherein various properties, including

5 triboelectric charges, are controlled by means as mentioned above. In such cases, the powder coating composition prepared in advance by mixing two or more powder coatings may be used, or as an alternative, the combination of two or more powder coatings according to

10 the present invention may be applied while blending the powder coatings. The means of coating are not particularly limited in the present invention, which may include such means as coating methods employing an electrostatic sprayer, fluidized bed coating methods, and

15 flame spraying methods. The amount of each of the powder coatings used in mixing colors may be suitably selected depending upon the desired hue to be obtained thereby.

In the present invention, a further preferable embodiment is that, in a case where a white powder coating

20 is used, at least one powder coating is a white one containing a white pigment, and the remaining powder coatings comprise no white pigments. The present invention is also concerned with a coating method for forming a coating film having visually homogeneous hue by

25 using the above combination of two or more powder

coatings. By using such a combination, a wide variety of hue from high to low lightness and chroma can be obtained depending upon the proportions of the each powder coatings combined.

5 Furthermore, the coating method of the present invention may comprise a previous step of applying a white under-coating on a substrate to be coated to give a substrate coated with the white coating. Then, onto the substrate, two or more powder coatings of which each color is different, wherein various kinds of physical properties, including differences in the triboelectric charges, are controlled within given ranges. In such a case, the colorants usable in white under-coatings include titanium oxide, calcium carbonate, barium sulfate, and 10 zinc flower, with a particular preference given to titanium oxide from the viewpoint of giving good covering ability. The white under-coatings are not particularly limited, and they may be powder coatings or they may be any of generally known white coatings such as aqueous 15 paints. In this method, it is preferred that two or more powder coatings, each of which contains no white pigments, may be used in combination. 20

By using the coating method of the present invention, coating films with a variety of visually homogeneous hue

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can be obtained on various substrates.

According to the present invention, since each of the powder coatings has an even triboelectric charge, it is possible to form a coating film having visually  
5 homogeneous hue by mixing powders of different tones of colors. Therefore, by preparing powders with several kinds of tones including primary colors, powders having all sorts of tones can be obtained, thereby making it unnecessary to stock powder coatings of numerous different  
10 tones as in cases of the conventional powder coatings.

#### EXAMPLES

The present invention will be explained in further detail by means of the following working examples  
15 illustrate without intending to limit the scope of the present invention thereto in any manner.

The powder coatings obtained in the following working examples are evaluated with respect to average particle sizes, triboelectric charges, true specific gravities,  
20 apparent densities, softening points, dielectric constants, and electric resistance by the following methods:

#### [Average Particle Size]

25 The average particle size is measured using a

Coulter-Multisizer (manufactured by Coulter K.K.).

[Triboelectric Charge]

First, a coating is applied using the coating machine  
5 and the coating object which are actually employed for  
coating, and working coating conditions (applied voltage  
and feeding amount of coating). A capacitor having an  
electric capacitance (C) of 0.047  $\mu$ F is connected between  
the coating object and the ground. The powder coating  
10 applied to the surface of the object is removed by  
air-blowing. The difference in voltage (V) which takes  
place in the capacitor owing to the efflux of electric  
charges ( $Q=CV$ ) caused by the removal of the powder coating  
is measured using an electrometer "TR 8411" (manufactured  
15 by Advantest Corporation). Also, the mass of the powder  
coating removed from the coating surface (M) is obtained  
by measuring the masses of the coating object before and  
after the removal of powder coating, and calculating the  
difference of the masses. From the values obtained, the  
20 triboelectric charge ( $Q/M$ ) is calculated.

[True Specific Gravity]

The true specific gravity is measured using a  
"Micromeritics Multivolume Pycnometer 1305" (manufactured  
25 by Shimadzu Corporation).

[Apparent Density]

The apparent density is measured using a powder tester (manufactured by Hosokawa Micron Corp.).

5 [Softening Point]

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The softening point is measured according to ASTM E28-67. The "softening point" used herein refers to the temperature corresponding to one-half of the height (h) of the S-shaped curve showing the relationship between the downward movement of a plunger (flow length) and temperature, when measured by using a capillary rheometer "SHIMADZU CFT-500 FLOW TESTER" of the "koka" type manufactured by Shimadzu Corporation in which a 1 cm<sup>3</sup> sample is extruded through a nozzle having a dice pore size of 1 mm and a length of 1 mm, while heating the sample so as to raise the temperature at a rate of 6°C/min and applying a load of 20 kg/cm<sup>2</sup> thereto with the plunger.

10  
15

[Dielectric Constant]

20 A pellet sample is prepared by pressing 5 g of each powder coating by applying a pressure of 0.4 t/cm<sup>2</sup> for 10 seconds using a press machine having a diameter of 59 mm. Using the pellet sample, the capacitance (Cp) at a frequency of 1 kHz is measured using an LCR meter

25 "HP16451B" (manufactured by Yokogawa-Hewlett-Packard

Ltd.). The dielectric constant is calculated by the following equation (1):

Dielectric constant =

5

$$\frac{(\text{Sample Thickness} \times C_p)}{(\text{Dielectric Constant in Vacuum} \times \text{Electrode Area})} \quad (1)$$

10

[Electric Resistance]

A pellet sample is prepared by pressing 5 g of each powder coating by applying a pressure of 0.4 t/cm<sup>2</sup> for 10 seconds using a press machine having a diameter of 59 mm.

15

Using the pellet sample, the conductance (G) at a frequency of 1 kHz is measured using an LCR meter "HP16451B" (manufactured by Yokogawa-Hewlett-Packard Ltd.). The electric resistance is calculated by the following equation (2):

20

Electric Resistance ( $\Omega \cdot \text{cm}^{-1}$ ) =

$$(1/G) \times \{(\text{Electrode Area})/(\text{Sample Thickness})\} \quad (2)$$

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Preparation Example 1-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a  
Super Mixer, and the resulting mixture was kneaded using a  
25 Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The  
kneaded mixture was cooled, the cooled product was  
pulverized using a pulverizer "PJM" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), and the obtained product  
was classified using a classifier "MDS" (manufactured by  
30 Nippon Pneumatic MFG, Co., Ltd.), to give a powder having  
an average particle size of 13  $\mu$ m. To 100 parts by weight  
of the resulting powder, 0.3 parts by weight of silica  
"AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were  
added, and the mixture was uniformly blended using a  
35 Henschel mixer, to give Powder Coating 1-1. The

triboelectric charge of Powder Coating 1-1 was  $-14.8 \mu\text{C/g}$ .

Preparation Example 1-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

- 25        The same procedures as in Preparation Example 1-1  
were carried out using the above components, to give a  
powder having an average particle size of  $13 \mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30    Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer, to give Powder Coating  
1-2. The triboelectric charge of Powder Coating 1-2 was  
 $-12.5 \mu\text{C/g}$ .



Preparation Example 1-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Carmine 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)             | 8 parts<br>by weight   |
| 15 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 1-1  
were carried out using the above components, to give a  
25 powder having an average particle size of 13  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer, to give Powder Coating  
30 1-3. The triboelectric charge of Powder Coating 1-3 was  
-10.0  $\mu\text{C/g}$ .

Preparation Example 1-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 94 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 10 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 15 | Benzoin   | 0.5 parts<br>by weight |

20 The same procedures as in Preparation Example 1-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
25 blended using a Henschel mixer, to give Powder Coating  
1-4. The triboelectric charge of Powder Coating 1-4 was  
-8.2  $\mu$ C/g.

Preparation Example 1-5 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 94 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 10 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 15 | Benzoin   | 0.5 parts<br>by weight |

20 The same procedures as in Preparation Example 1-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
25 blended using a Henschel mixer, to give Powder Coating  
1-5. The triboelectric charge of Powder Coating 1-5 was  
-17.3  $\mu\text{C/g}$ .

Preparation Example 1-6 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 1-1  
were carried out using the above components, to give a  
25 powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer, to give Powder Coating  
30 1-6. The triboelectric charge of Powder Coating 1-6 was  
-13.6  $\mu$ C/g.

Example A-1

50 parts by weight of Powder Coating 1-1 and 50 parts  
35 by weight of Powder Coating 1-2 were blended using a

Henschel mixer. The resulting mixture was applied to a degreased steel substrate using an electrostatic sprayer, and the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The difference in the triboelectric charges between the powder coatings blended is shown in Table 1.

Example A-2

50 parts by weight of Powder Coating 1-1 and 50 parts by weight of Powder Coating 1-3 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example A-1 to form a coating film. The formed coating film was also visually homogeneous blue. The difference in the triboelectric charges between the powder coatings blended is shown in Table 1.

Comparative Example a-1

50 parts by weight of Powder Coating 1-1 and 50 parts by weight of Powder Coating 1-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example A-1 to form a coating film. The coating state of each of the powder coatings was not even, so that the resulting

coating film had an uneven thickness and showed color separation between cyan and magenta. The difference in the triboelectric charges between the powder coatings blended is shown in Table 1.

5

Comparative Example a-2

50 parts by weight of Powder Coating 1-3 and 50 parts by weight of Powder Coating 1-5 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example A-1 to form a coating film. The coating state of each of the powder coatings was not even, so that the resulting coating film had an uneven thickness and showed color separation between cyan and magenta. The difference in the triboelectric charges between the powder coatings blended is shown in Table 1.

10

15

Comparative Example a-3

50 parts by weight of Powder Coating 1-4 and 50 parts by weight of Powder Coating 1-6 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example A-1 to form a coating film. The coating state of each of the powder coatings was not even, so that the resulting coating film had an uneven thickness and showed color

20

25

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[illegible]

T a b l e 1

|                            |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g) |
|----------------------------|--|---|
| Example A-1                | Powder<br>Coating (1-1)<br>Powder<br>Coating (1-2) | 2.3   |
| Example A-2                | Powder<br>Coating (1-1)<br>Powder<br>Coating (1-3) | 4.8   |
| Comparative<br>Example a-1 | Powder<br>Coating (1-1)<br>Powder<br>Coating (1-4) | 6.6   |
| Comparative<br>Example a-2 | Powder<br>Coating (1-1)<br>Powder<br>Coating (1-5) | 7.3   |
| Comparative<br>Example a-3 | Powder<br>Coating (1-1)<br>Powder<br>Coating (1-6) | 5.4   |



Preparation Example 2-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 40 parts<br>by weight  |
| 15 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 12  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 2-1. The

triboelectric charge of Powder Coating 2-1 was  $-15.9 \mu\text{C/g}$  and the true specific gravity was  $1.4820 \text{ g/cc}$ .

Preparation Example 2-2 of Powder Coating

|    |  |                        |
|----|--|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = $32.5 \text{ mg KOH/g}$ ) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = $65.8 \text{ mg KOH/g}$ ) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)  | 6 parts<br>by weight   |
|    | Zinc Oxide (manufactured by<br>Sakai Chemical Industry Co., Ltd.)  | 30 parts<br>by weight  |
| 20 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)                         | 8 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)  | 1 part<br>by weight    |
|    | Benzoin  | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 2-1 were carried out using the above components, to give a powder having an average particle size of  $12 \mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 2-2. The triboelectric charge of Powder Coating 2-2 was  $-12.9 \mu\text{C/g}$ , and the true specific gravity was  $1.4532 \text{ g/cc}$ .

Preparation Example 2-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Zinc Oxide (manufactured by<br>Sakai Chemical Industry Co., Ltd.)   | 30 parts<br>by weight  |
| 25 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
| 30 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 35 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 2-1 were carried out using the above components, to give a powder having an average particle size of 12  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 2-3. The triboelectric charge of Powder Coating 2-3 was -15.3  $\mu\text{C/g}$ , and the true specific gravity was 1.4474 g/cc.

Preparation Example 2-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Zinc Oxide (manufactured by<br>Sakai Chemical Industry Co., Ltd.)                                     | 30 parts<br>by weight  |
| 15 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

25

The same procedures as in Preparation Example 2-1 were carried out using the above components, to give a powder having an average particle size of 12  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 2-4. The triboelectric charge of Powder Coating 2-4 was -14.6  $\mu$ C/g, and the true specific gravity was 1.4743 g/cc.

35

Preparation Example 2-5 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 15 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 2-1  
were carried out using the above components, to give a  
powder having an average particle size of 12  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer, to give Powder Coating  
2-5. The triboelectric charge of Powder Coating 2-5 was  
-15.3  $\mu\text{C/g}$ , and the true specific gravity was 1.3233 g/cc.

Preparation Example 2-6 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 2-1 were carried out using the above components, to give a powder having an average particle size of 12  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 2-6. The triboelectric charge of Powder Coating 2-6 was -17.2  $\mu\text{C/g}$ , and the true specific gravity was 1.3039 g/cc.

Example B-1

20 parts by weight of Powder Coating 2-1, 40 parts by weight of Powder Coating 2-2, and 40 parts by weight of

Powder Coating 2-3 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate using an electrostatic sprayer, and the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The maximum differences in the triboelectric charges and in the true specific gravities between the powder coatings blended are respectively shown in Table 2.

Example B-2

20 parts by weight of Powder Coating 2-1, 40 parts by weight of Powder Coating 2-3, and 40 parts by weight of Powder Coating 2-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example B-1 to form a coating film. The formed coating film was visually homogeneous green. The maximum differences in the triboelectric charges and in the true specific gravities between the powder coatings blended are respectively shown in Table 2.

Example B-3

20 parts by weight of Powder Coating 2-1, 40 parts by weight of Powder Coating 2-2, and 40 parts by weight of

Powder Coating 2-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example B-1 to form a coating film. The formed coating film was visually homogeneous blue. The maximum differences in the triboelectric charges and in the true specific gravities between the powder coatings blended are respectively shown in Table 2.

10 Comparative Example b-1

20 parts by weight of Powder Coating 2-1, 40 parts by weight of Powder Coating 2-3, and 40 parts by weight of Powder Coating 2-5 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example B-1 to form a coating film. The formed coating film was visually inhomogeneous red. The maximum differences in the triboelectric charges and in the true specific gravities between the powder coatings blended are respectively shown in Table 2.

Comparative Example b-2

20 parts by weight of Powder Coating 2-1, 40 parts by weight of Powder Coating 2-4, and 40 parts by weight of Powder Coating 2-6 were blended using a Henschel mixer.



The resulting mixture was applied to a degreased steel substrate in the same manner as in Example B-1 to form a coating film. The formed coating film was visually inhomogeneous green. The maximum differences in the triboelectric charges and in the true specific gravities between the powder coatings blended are respectively shown in Table 2.

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Table 2

|                            |   | Difference in<br>Triboelectric<br>Charges<br>( $\mu C / g$ ) | Difference in<br>True Specific<br>Gravities<br>( $g / c c$ ) |
|----------------------------|---|--|--|
| Example B-1                | Powder<br>Coating (2-1)<br>Powder<br>Coating (2-2)<br>Powder<br>Coating (2-3) | 3.0  | 0.0346   |
| Example B-2                | Powder<br>Coating (2-1)<br>Powder<br>Coating (2-3)<br>Powder<br>Coating (2-4) | 1.3  | 0.0346   |
| Example B-3                | Powder<br>Coating (2-1)<br>Powder<br>Coating (2-2)<br>Powder<br>Coating (2-4) | 3.0  | 0.0288   |
| Comparative<br>Example b-1 | Powder<br>Coating (2-1)<br>Powder<br>Coating (2-3)<br>Powder<br>Coating (2-5) | 0.6  | 0.1587   |
| Comparative<br>Example b-2 | Powder<br>Coating (2-1)<br>Powder<br>Coating (2-4)<br>Powder<br>Coating (2-6) | 2.6  | 0.1781   |

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Preparation Example 3-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                 | 94 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                 | 6 parts<br>by weight   |
| 10 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.) | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                   | 1 part<br>by weight    |
| 15 | Benzoin   | 0.5 parts<br>by weight |

20 The above components were previously blended using a  
Super Mixer, and the resulting mixture was kneaded using a  
Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The  
kneaded mixture was cooled, the cooled product was  
pulverized using a pulverizer "PJM" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), and the obtained product  
25 was classified using a classifier "MDS" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), to give a powder having  
an average particle size of 13  $\mu\text{m}$ . To 100 parts by weight  
of the resulting powder, 0.3 parts by weight of silica  
"AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were  
30 added, and the mixture was uniformly blended using a  
Henschel mixer, to give Powder Coating 3-1. The  
triboelectric charge of Powder Coating 3-1 was  $-12.1 \mu\text{C/g}$   
and the apparent density was 0.405 g/cc.

Preparation Examples 3-2 to 3-6 of Powder Coatings

|    |  |                        |
|----|--|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                | 94 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.) | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                  | 1 part<br>by weight    |
| 25 | Benzoin  | 0.5 parts<br>by weight |

20 The same procedures as in Preparation Example 3-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, the amount of  
silica "AEROSIL R972" (manufactured by Nippon Aerosil  
Ltd.) were respectively varied to 0.3 parts by weight, 0.5  
25 parts by weight, 0.6 parts by weight, and 0.7 parts by  
weight, and each resulting mixture was uniformly blended  
using a Henschel mixer, to give the respective Powder  
Coating 3-2, Powder Coating 3-3, Powder Coating 3-4,  
Powder Coating 3-5, and Powder Coating 3-6. The  
30 triboelectric charges of each of Powder Coatings were as  
follows. Powder Coating 3-2 had a triboelectric charge of  
-7.6  $\mu$ C/g, Powder Coating 3-3 a triboelectric charge of  
-8.2  $\mu$ C/g, Powder Coating 3-4 a triboelectric charge of

-9.3  $\mu\text{C/g}$ , Powder Coating 3-5 a triboelectric charge of  
-9.8  $\mu\text{C/g}$ , and Powder Coating 3-6 a triboelectric charge  
of -10.7  $\mu\text{C/g}$ . The apparent densities of each Powder  
Coating were as follows. Powder Coating 3-2 had an  
5 apparent density of 0.382 g/cc, Powder Coating 3-3 an  
apparent density of 0.401 g/cc, Powder Coating 3-4 an  
apparent density of 0.420 g/cc, Powder Coating 3-5 an  
apparent density of 0.424/cc, and Powder Coating 3-6 an  
apparent density of 0.430 g/cc.

10

Example C-1

50 parts by weight of Powder Coating 3-1 and 50 parts  
by weight of Powder Coating 3-3 were blended using a  
Henschel mixer. The resulting mixture was applied to a  
15 degreased steel substrate using an electrostatic sprayer,  
and the coated steel substrate was stoved at 180°C for  
20 minutes to form a coating film. The formed coating  
film was visually homogeneous blue. The differences in  
the triboelectric charges and in the apparent densities  
20 between the powder coatings blended are respectively shown  
in Table 3.

Example C-2

50 parts by weight of Powder Coating 3-1 and 50 parts  
25 by weight of Powder Coating 3-4 were blended using a

Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example C-1 to form a coating film. The formed coating film also was visually homogeneous blue. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 3.

Example C-3

50 parts by weight of Powder Coating 3-1 and 50 parts by weight of Powder Coating 3-5 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example C-1 to form a coating film. The formed coating also was also visually homogeneous blue. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 3.

Comparative Example c-1

50 parts by weight of Powder Coating 3-1 and 50 parts by weight of Powder Coating 3-2 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example C-1 to form a coating film. The coating state of each of

the powder coatings was slightly uneven, so that the resulting coating film had an uneven thickness, and also showed color separation between cyan and magenta. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 3.

Comparative Example c-2

50 parts by weight of Powder Coating 3-1 and 50 parts by weight of Powder Coating 3-6 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example C-1 to form a coating film. The coating state of each of the powder coatings was visibly uneven, so that the resulting coating film had an uneven thickness, and also showed distinct color separation between cyan and magenta. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 3.

T a b l e 3

|                            |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu C / g$ ) | Difference in<br>Apparent<br>Densities<br>( $g / c c$ ) |
|----------------------------|--|--|---|
| Example C-1                | Powder<br>Coating (3-1)<br>Powder<br>Coating (3-3) | 3.9  | 0.004   |
| Example C-2                | Powder<br>Coating (3-1)<br>Powder<br>Coating (3-4) | 2.8  | 0.015   |
| Example C-3                | Powder<br>Coating (3-1)<br>Powder<br>Coating (3-5) | 2.3  | 0.019   |
| Comparative<br>Example c-1 | Powder<br>Coating (3-1)<br>Powder<br>Coating (3-2) | 4.5  | 0.023   |
| Comparative<br>Example c-2 | Powder<br>Coating (3-1)<br>Powder<br>Coating (3-6) | 1.4  | 0.025   |

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Preparation Example 4-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8700,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                 | 94 parts<br>by weight  |
| 5  | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                 | 6 parts<br>by weight   |
| 10 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.) | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                   | 1 part<br>by weight    |
| 15 | Benzoin   | 0.5 parts<br>by weight |

20 The above components were previously blended using a  
Super Mixer, and the resulting mixture was kneaded using a  
Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The  
kneaded mixture was cooled, the cooled product was  
pulverized using a pulverizer "PJM" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), and the obtained product  
25 was classified using a classifier "MDS" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), to give a powder having  
an average particle size of 13  $\mu\text{m}$ . To 100 parts by weight  
of the resulting powder, 0.3 parts by weight of silica  
"AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were  
30 added, and the mixture was uniformly blended using a  
Henschel mixer, to give Powder Coating 4-1. The  
triboelectric charge of Powder Coating 4-1 was  $-12.1 \mu\text{C/g}$ ,  
and the softening point was  $114.1^\circ\text{C}$ .

Preparation Example 4-2 of Powder Coating

|    |  |                        |
|----|--|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                | 94 parts<br>by weight  |
| 5  | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                | 6 parts<br>by weight   |
| 10 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.) | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                  | 1 part<br>by weight    |
| 15 | Benzoin  | 0.5 parts<br>by weight |

20 The same procedures as in Preparation Example 4-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
25 blended using a Henschel mixer, to give Powder Coating  
4-2. The triboelectric charge of Powder Coating 4-2 was  
-8.2  $\mu$ C/g, and the softening point was 111.6°C.

Preparation Example 4-3 of Powder Coating

|    |  |                        |
|----|--|------------------------|
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester,<br>Co., Ltd.)               | 47 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                | 47 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                | 6 parts<br>by weight   |
|    | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.) | 8 parts<br>by weight   |
| 15 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                  | 1 part<br>by weight    |
| 20 | Benzoin  | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 4-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer, to give Powder Coating  
4-3. The triboelectric charge of Powder Coating 4-3 was  
-9.8  $\mu\text{C/g}$ , and the softening point was 109.2°C.

Example D-1

50 parts by weight of Powder Coating 4-1 and 50 parts  
by weight of Powder Coating 4-2 were blended using a

Henschel mixer. The resulting mixture was applied to a degreased steel substrate using an electrostatic sprayer, and the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The differences in the triboelectric charges and in the softening points between the powder coatings blended are respectively shown in Table 4.

10 Example D-2

50 parts by weight of Powder Coating 4-1 and 50 parts by weight of Powder Coating 4-3 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example D-1 to form a coating film. The formed coating film was also visually homogeneous blue. The differences in the triboelectric charges and in the softening points between the powder coatings blended are respectively shown in Table 4.

T a b l e 4

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g ) | Difference in<br>Softening<br>Points<br>( $^{\circ}$ C) |
|-------------|--|--|---|
| Example D-1 | Powder<br>Coating (4-1)<br>Powder<br>Coating (4-2) | 3.9  | 2.5   |
| Example D-2 | Powder<br>Coating (4-1)<br>Powder<br>Coating (4-3) | 2.3  | 4.9   |

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Preparation Example 5-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 40 parts<br>by weight  |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 13  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 5-1 with a white

color.

Preparation Example 5-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

- 25        The same procedures as in Preparation Example 5-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30        Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 5-2.  
The triboelectric charge of Powder Coating 5-2 was  
-16.8  $\mu\text{C/g}$ .

Preparation Example 5-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 5-1 were carried out using the above components, to give a powder having an average particle size of 13  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 5-3.

The triboelectric charge of Powder Coating 5-3 was -13.6  $\mu\text{C/g}$ .



Preparation Example 5-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 5-1 were carried out using the above components, to give a powder having an average particle size of 13  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 5-4.

The triboelectric charge of Powder Coating 5-4 was -13.8  $\mu\text{C/g}$ .

Preparation Example 5-5 of Powder Coating

The same procedures as in Preparation Example 5-2 were carried out except that the amount of Carmine 6B ("SUMIKAPRINT CARMINE 6BC" manufactured by Sumitomo Chemical Co., Ltd.) was changed to 10 parts by weight, to give Powder Coating 5-5. The triboelectric charge of Powder Coating 5-5 was  $-8.1 \mu\text{C/g}$ .

Example E-1

10        100 parts by weight of Powder Coating 5-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 15        5-2 and 50 parts by weight of Powder Coating 5-3 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at  $180^{\circ}\text{C}$  for 20 minutes to form a coating film. The formed coating film was 20        visually homogeneous red. The difference in the triboelectric charges between the powder coatings blended is shown in Table 5.

Example E-2

25        100 parts by weight of Powder Coating 5-1 were

applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 5-3 and 50 parts by weight of Powder Coating 5-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous green. The difference in the triboelectric charges between the powder coatings blended is shown in Table 5.

#### Example E-3

100 parts by weight of Powder Coating 5-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 5-2 and 50 parts by weight of Powder Coating 5-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The difference in the

triboelectric charges between the powder coatings blended is shown in Table 5.

Comparative Example e-1

5           100 parts by weight of Powder Coating 5-1 were  
applied to a degreased steel substrate using an  
electrostatic sprayer to form a white coating.  
Thereafter, the voltage was increased, and a mixture  
prepared by blending 50 parts by weight of Powder Coating  
10   5-3 and 50 parts by weight of Powder Coating 5-5 using a  
Henschel mixer was applied using an electrostatic sprayer  
to the substrate with a white coating. Subsequently, the  
coated steel substrate was stoved at 180°C for 20 minutes  
to form a coating film. The resulting coating film had an  
15   uneven thickness, and showed uneven density of magenta and  
yellow. The difference in the triboelectric charges  
between the powder coatings blended is shown in Table 5.

T a b l e 5

|                            |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g ) |
|----------------------------|--|--|
| Example E-1                | Powder<br>Coating (5-2)<br>Powder<br>Coating (5-3) | 3.2  |
| Example E-2                | Powder<br>Coating (5-3)<br>Powder<br>Coating (5-4) | 0.2  |
| Example E-3                | Powder<br>Coating (5-2)<br>Powder<br>Coating (5-4) | 3.0  |
| Comparative<br>Example e-1 | Powder<br>Coating (5-3)<br>Powder<br>Coating (5-5) | 5.5  |

Preparation Example 6-1 of Powder Coating

|    |  |                        |
|----|--|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)            | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd.)            | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                            | 6 parts<br>by weight   |
| 15 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.) | 40 parts<br>by weight  |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                              | 1 part<br>by weight    |
| 20 | Benzoin  | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 10  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 6-1 having a white

color.

Preparation Example 6-2 of Powder Coating

|    |  |                        |
|----|--|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.) | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                  | 1 part<br>by weight    |
|    | Benzoin  | 0.5 parts<br>by weight |

- 25        The same procedures as in Preparation Example 6-1  
were carried out using the above components, to give a  
powder having an average particle size of 10  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30    Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 6-2.  
The triboelectric charge of Powder Coating 6-2 was  
-14.1  $\mu$ C/g, and the apparent density was 0.10 g/cc.

Preparation Example 6-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                                       | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                                       | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

- The same procedures as in Preparation Example 6-1 were carried out using the above components, to give a powder having an average particle size of 10  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 6-3.
- The triboelectric charge of Powder Coating 6-3 was -17.7  $\mu$ C/g, and the apparent density was 0.409 g/cc.



Preparation Example 6-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                 | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd.)                 | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)                                 | 6 parts<br>by weight   |
| 15 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.) | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)                                   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 6-1  
were carried out using the above components, to give a  
powder having an average particle size of 10  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 6-4.  
The triboelectric charge of Powder Coating 6-4 was  
-15.9  $\mu$ C/g, and the apparent density was 0.410 g/cc.

Preparation Example 6-5 of Powder Coating

The same procedures as in Preparation Example 6-2  
were carried out except that the amount of silica "AEROSIL

R972" (manufactured by Nippon Aerosil Ltd.) was changed to 0.15 parts by weight, to give Powder Coating 6-5. The triboelectric charge of Powder Coating 6-5 was  $-13.0 \mu\text{C/g}$ , and the apparent density was  $0.385 \text{ g/cc}$ .

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Preparation Example 6-6 of Powder Coating

The same procedures as in Preparation Example 6-3 were carried out except that the amount of silica "AEROSIL R972" (Nippon Aerosil Ltd.) was changed to 0.6 parts by weight, to give Powder Coating 6-6. The triboelectric charge of Powder Coating 6-6 was  $-18.2 \mu\text{C/g}$ , and the apparent density was  $0.436 \text{ g/cc}$ .

10

Example F-1

100 parts by weight of Powder Coating 6-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 6-2 and 50 parts by weight of Powder Coating 6-3 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at  $180^\circ\text{C}$  for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The differences in the

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triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 6.

5     Example F-2

100 parts by weight of Powder Coating 6-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

10     Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 6-3 and 50 parts by weight of Powder Coating 6-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous green. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 6.

20

Example F-3

100 parts by weight of Powder Coating 6-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

25     Thereafter, the voltage was increased, and a mixture

prepared by blending 50 parts by weight of Powder Coating 6-2 and 50 parts by weight of Powder Coating 6-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The differences in the triboelectric charges and in the apparent densities between the powder coatings blended are respectively shown in Table 6.

Comparative Example f-1

100 parts by weight of Powder Coating 6-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 6-3 and 50 parts by weight of Powder Coating 6-5 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The resulting coating film had an uneven thickness and also showed color separation between magenta and yellow. The differences in the triboelectric charges and in the apparent densities between the powder

coatings blended are respectively shown in Table 6.

Comparative Example f-2

100 parts by weight of Powder Coating 6-1 were  
5 applied to a degreased steel substrate using an  
electrostatic sprayer to form a white coating.  
Thereafter, the voltage was increased, and a mixture  
prepared by blending 50 parts by weight of Powder Coating  
6-4 and 50 parts by weight of Powder Coating 6-6 using a  
10 Henschel mixer was applied using an electrostatic sprayer  
to the substrate with a white coating. Subsequently, the  
coated steel substrate was stoved at 180°C for 20 minutes  
to form a coating film. The resulting coating film had an  
uneven thickness and also showed color separation between  
15 yellow and cyan. The differences in the triboelectric  
charges and in the apparent densities between the powder  
coatings blended are respectively shown in Table 6.

T a b l e 6

|                            |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu C / g$ ) | Difference in<br>Apparent<br>Densities<br>( $g / c c$ ) |
|----------------------------|--|--|---|
| Example F-1                | Powder<br>Coating (6-2)<br>Powder<br>Coating (6-3) | 3.6  | 0.001   |
| Example F-2                | Powder<br>Coating (6-3)<br>Powder<br>Coating (6-4) | 1.8  | 0.001   |
| Example F-3                | Powder<br>Coating (6-2)<br>Powder<br>Coating (6-4) | 1.8  | 0.000   |
| Comparative<br>Example f-1 | Powder<br>Coating (6-3)<br>Powder<br>Coating (6-5) | 4.7  | 0.024   |
| Comparative<br>Example f-2 | Powder<br>Coating (6-4)<br>Powder<br>Coating (6-6) | 2.3  | 0.026   |

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Preparation Example 7-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 40 parts<br>by weight  |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 13  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 7-1 with a white

color.

Preparation Example 7-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

- 25        The same procedures as in Preparation Example 7-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30    Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 7-2.  
The triboelectric charge of Powder Coating 7-2 was  
-16.8  $\mu$ C/g, and the softening point was 110°C.



Preparation Example 7-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 94 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 10 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 15 | Benzoin   | 0.5 parts<br>by weight |

20 The same procedures as in Preparation Example 7-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
25 blended using a Henschel mixer to give Powder Coating 7-3.  
The triboelectric charge of Powder Coating 7-3 was  
-16.8  $\mu\text{C/g}$ , and the softening point was 107°C.

Preparation Example 7-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | 5 TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

25 The same procedures as in Preparation Example 7-1  
were carried out using the above components, to give a  
powder having an average particle size of 13  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 7-4.  
30 The triboelectric charge of Powder Coating 7-4 was  
-13.8  $\mu$ C/g, and the softening point was 110°C.

Example G-1

35 100 parts by weight of Powder Coating 7-1 were  
applied to a degreased steel substrate using an

electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 7-2 and 50 parts by weight of Powder Coating 7-3 using a

5 Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The differences in the  
10 triboelectric charges and in the softening points between the powder coatings blended are respectively shown in Table 7.

Example G-2

15 100 parts by weight of Powder Coating 7-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating  
20 7-3 and 50 parts by weight of Powder Coating 7-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was  
25 visually homogeneous green. The differences in the

triboelectric charges and in the softening points between the powder coatings blended are respectively shown in Table 7.

5     Example G-3

100 parts by weight of Powder Coating 7-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 7-2 and 50 parts by weight of Powder Coating 7-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The differences in the triboelectric charges and in the softening points between the powder coatings blended are respectively shown in Table 7.

20

Table 7

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g) | Difference in<br>Softening<br>Points<br>( $^{\circ}$ C) |
|-------------|--|---|---|
| Example G-1 | Powder<br>Coating (7-2)<br>Powder<br>Coating (7-3) | 0.0   | 3.0   |
| Example G-2 | Powder<br>Coating (7-3)<br>Powder<br>Coating (7-4) | 3.0   | 3.0   |
| Example G-3 | Powder<br>Coating (7-2)<br>Powder<br>Coating (7-4) | 3.0   | 0.0   |

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Preparation Example 8-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 40 parts<br>by weight  |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 8  $\mu$ m. To 100 parts by weight of the resulting powder, 0.45 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 8-1 with a white

color.

Preparation Example 8-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

- 25        The same procedures as in Preparation Example 8-1  
were carried out using the above components, to give a  
powder having an average particle size of 8  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.45 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
30        blended using a Henschel mixer to give Powder Coating 8-2.  
The triboelectric charge of Powder Coating 8-2 was  
-16.8  $\mu$ C/g, and the dielectric constant was 2.85.

Preparation Example 8-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

- The same procedures as in Preparation Example 8-1 were carried out using the above components, to give a powder having an average particle size of 8  $\mu$ m. To 100 parts by weight of the resulting powder, 0.45 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 8-3.
- The triboelectric charge of Powder Coating 8-3 was -18.1  $\mu$ C/g, and the dielectric constant was 2.78.



Preparation Example 8-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 5  | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 8-1  
were carried out using the above components, to give a  
powder having an average particle size of 8  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.45 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 8-4.  
The triboelectric charge of Powder Coating 8-4 was  
-18.2  $\mu$ C/g, and the dielectric constant was 2.76.

Example H-1

100 parts by weight of Powder Coating 8-1 were  
applied to a degreased steel substrate using an

electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 8-2 and 50 parts by weight of Powder Coating 8-3 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The differences in the triboelectric charges and in the dielectric constants between the powder coatings blended are respectively shown in Table 8.

#### Example H-2

100 parts by weight of Powder Coating 8-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 8-3 and 50 parts by weight of Powder Coating 8-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous green. The differences in the

triboelectric charges and in the dielectric constants between the powder coatings blended are respectively shown in Table 8.

5     Example H-3

100 parts by weight of Powder Coating 8-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 8-2 and 50 parts by weight of Powder Coating 8-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The differences in the triboelectric charges and in the dielectric constants between the powder coatings blended are respectively shown in Table 8.

T a b l e 8

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g ) | Difference in<br>Dielectric<br>Constants |
|-------------|--|--|--|
| Example H-1 | Powder<br>Coating (8-2)<br>Powder<br>Coating (8-3) | 1.3  | 0.07                                     |
| Example H-2 | Powder<br>Coating (8-3)<br>Powder<br>Coating (8-4) | 0.1  | 0.02                                     |
| Example H-3 | Powder<br>Coating (8-2)<br>Powder<br>Coating (8-4) | 1.4  | 0.09                                     |

Preparation Example 9-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 40 parts<br>by weight  |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

The above components were previously blended using a Super Mixer, and the resulting mixture was kneaded using a Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The kneaded mixture was cooled, the cooled product was pulverized using a pulverizer "PJM" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), and the obtained product was classified using a classifier "MDS" (manufactured by Nippon Pneumatic MFG, Co., Ltd.), to give a powder having an average particle size of 15  $\mu$ m. To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer, to give Powder Coating 9-1 with a white

color.

Preparation Example 9-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 15 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 79 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Carmine 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)             | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

25        The same procedures as in Preparation Example 9-1  
were carried out using the above components, to give a  
powder having an average particle size of 15  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30    Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating 9-2.  
The triboelectric charge of Powder Coating 9-2 was  
-12.8  $\mu\text{C/g}$ , and the electric resistance was  
4.7  $\times 10^{11} \Omega \cdot \text{cm}^{-1}$ .

35

Preparation Example 9-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 9-1 were carried out using the above components, to give a powder having an average particle size of 15  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 9-3.

The triboelectric charge of Powder Coating 9-3 was -11.8  $\mu\text{C/g}$ , and the electric resistance was  $3.5 \times 10^{11} \Omega \cdot \text{cm}^{-1}$ .

Preparation Example 9-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 5 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 9-1 were carried out using the above components, to give a powder having an average particle size of 15  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 9-4.

The triboelectric charge of Powder Coating 9-4 was  $-12.4 \mu\text{C/g}$ , and the electric resistance was  $5.5 \times 10^{11} \Omega \cdot \text{cm}^{-1}$ .

Example I-1

100 parts by weight of Powder Coating 9-1 were



applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 9-2 and 50 parts by weight of Powder Coating 9-3 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The difference in the triboelectric charges between the powder coatings blended and the ratio of electric resistance of Powder Coating 9-2 to Powder Coating 9-3 are shown in Table 9.

Example I-2

100 parts by weight of Powder Coating 9-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating. Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 9-3 and 50 parts by weight of Powder Coating 9-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was

visually homogeneous green. The difference in the triboelectric charges between the powder coatings blended and the ratio of electric resistance of Powder Coating 9-4 to Powder Coating 9-3 are shown in Table 9.

5

Example I-3

100 parts by weight of Powder Coating 9-1 were applied to a degreased steel substrate using an electrostatic sprayer to form a white coating.

10 Thereafter, the voltage was increased, and a mixture prepared by blending 50 parts by weight of Powder Coating 9-2 and 50 parts by weight of Powder Coating 9-4 using a Henschel mixer was applied using an electrostatic sprayer to the substrate with a white coating. Subsequently, the  
15 coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous blue. The difference in the triboelectric charges between the powder coatings blended and the ratio of electric resistance of Powder Coating 9-4  
20 to Powder Coating 9-2 are shown in Table 9.

T a b l e 9

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu$ C / g ) | Ratio of<br>Electric<br>Resistances |
|-------------|--|--|-------------------------------------|
| Example I-1 | Powder<br>Coating (9-2)<br>Powder<br>Coating (9-3) | 1.0  | 1.3                                 |
| Example I-2 | Powder<br>Coating (9-3)<br>Powder<br>Coating (9-4) | 0.6  | 1.6                                 |
| Example I-3 | Powder<br>Coating (9-2)<br>Powder<br>Coating (9-4) | 0.4  | 1.2                                 |

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Preparation Example 10-1 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Titanium Oxide ("TIPAQUE CR-90,"<br>manufactured by ISHIHARA SANGYO<br>KAISHA, LTD.)                  | 20 parts<br>by weight  |
|    | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

25 The above components were previously blended using a  
Super Mixer, and the resulting mixture was kneaded using a  
Buss Ko-kneader (manufactured by Buss (Japan) Ltd.). The  
kneaded mixture was cooled, the cooled product was  
pulverized using a pulverizer "PJM" (manufactured by  
Nippon Pneumatic MFG, Co., Ltd.), and the obtained product  
was classified using a classifier "MDS" (manufactured by  
30 Nippon Pneumatic MFG, Co., Ltd.), to give a powder having  
an average particle size of 12  $\mu$ m. To 100 parts by weight  
of the resulting powder, 0.3 parts by weight of silica  
"AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were  
added, and the mixture was uniformly blended using a  
35 Henschel mixer, to give Powder Coating 10-1 with a white

color. The triboelectric charge of Powder Coating 10-1 was -15.9  $\mu\text{C/g}$ , the apparent density was 0.416 g/cc, and the softening point was 110°C.

5     Preparation Example 10-2 of Powder Coating

|    |   |                        |
|----|---|------------------------|
|    | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Carmin 6B ("SUMIKAPRINT CARMINE 6BC,"<br>manufactured by Sumitomo Chemical<br>Co., Ltd.)              | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 25 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 10-1 were carried out using the above components, to give a powder having an average particle size of 12  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 10-2. The triboelectric charge of Powder Coating 10-2 was -15.3  $\mu\text{C/g}$ , the apparent density was 0.412 g/cc, and the

softening point was 111°C.

Preparation Example 10-3 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g)           | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g)           | 54 parts<br>by weight  |
|    | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 15 | Dis-azo Yellow ("PIGMENT YELLOW ECY-210,"<br>manufactured by Dainichiseika Color &<br>Chemicals MFG. Co., Ltd.) | 8 parts<br>by weight   |
| 20 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
|    | Benzoin   | 0.5 parts<br>by weight |

25           The same procedures as in Preparation Example 10-1  
were carried out using the above components, to give a  
powder having an average particle size of 12  $\mu$ m. To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
30   Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating  
10-3. The triboelectric charge of Powder Coating 10-3 was  
-17.2  $\mu$ C/g, the apparent density was 0.411 g/cc, and the  
softening point was 112°C.

Preparation Example 10-4 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
| 10 | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 15 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
| 20 | Copper Phthalocyanine<br>("CYANINE BLUE-KRS," manufactured by<br>SANYO COLOR WORKS, LTD.)             | 6 parts<br>by weight   |
| 25 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 30 | Benzoin   | 0.5 parts<br>by weight |

The same procedures as in Preparation Example 10-1 were carried out using the above components, to give a powder having an average particle size of 12  $\mu\text{m}$ . To 100 parts by weight of the resulting powder, 0.3 parts by weight of silica "AEROSIL R972" (manufactured by Nippon Aerosil Ltd.) were added, and the mixture was uniformly blended using a Henschel mixer to give Powder Coating 10-4. The triboelectric charge of Powder Coating 10-4 was -16.5  $\mu\text{C/g}$ , the apparent density was 0.412 g/cc, and the softening point was 110°C.

Preparation Example 10-5 of Powder Coating

|    |   |                        |
|----|---|------------------------|
| 5  | Polyester Resin ("ER-8107,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 32.5 mg KOH/g) | 40 parts<br>by weight  |
|    | Polyester Resin ("ER-8100,"<br>manufactured by Nippon Ester<br>Co., Ltd., acid value = 65.8 mg KOH/g) | 54 parts<br>by weight  |
| 10 | TGIC ("ARALDITE PT810,"<br>manufactured by Ciba Geigy AG)   | 6 parts<br>by weight   |
|    | Carbon Black ("MOGAL L,"<br>manufactured by Cabot Japan K.K.)   | 8 parts<br>by weight   |
| 15 | Levelling Agent ("ACRONAL 4F,"<br>manufactured by BASF)   | 1 part<br>by weight    |
| 20 | Benzoin   | 0.5 parts<br>by weight |

25 The same procedures as in Preparation Example 10-1  
were carried out using the above components, to give a  
powder having an average particle size of 12  $\mu\text{m}$ . To  
100 parts by weight of the resulting powder, 0.3 parts by  
weight of silica "AEROSIL R972" (manufactured by Nippon  
Aerosil Ltd.) were added, and the mixture was uniformly  
blended using a Henschel mixer to give Powder Coating  
10-5. The triboelectric charge of Powder Coating 10-5 was  
30 -14.5  $\mu\text{C/g}$ , the apparent density was 0.413 g/cc, and the  
softening point was 113°C.

Preparation Example 10-6 of Powder Coating

35 The same procedures as in Preparation Example 10-2



were carried out except that the amount of Carmine 6B was changed to 6 parts by weight and titanium oxide ("TIPAQUE CR-90," manufactured by ISHIHARA SANGYO KAISHA, LTD.) was added in an amount of 10 parts by weight, to give Powder Coating 10-6. The triboelectric charge of Powder Coating 10-6 was -13.8  $\mu\text{C/g}$ , the apparent density was 0.415 g/cc, and the softening point was 114°C.

Preparation Example 10-7 of Powder Coating

The same procedures as in Preparation Example 10-3 were carried out except that the amount of dis-azo yellow was changed to 6 parts by weight and the amount of titanium oxide ("TIPAQUE CR-90," manufactured by ISHIHARA SANGYO KAISHA, LTD.) was added in an amount of 10 parts by weight, to give Powder Coating 10-7. The triboelectric charge of Powder Coating 10-7 was -16.1  $\mu\text{C/g}$ , the apparent density was 0.414 g/cc, and the softening point was 113°C.

Preparation Example 10-8 of Powder Coating

The same procedures as in Preparation Example 10-4 were carried out except that the amount of copper phthalocyanine was changed to 4.5 parts by weight and the amount of titanium oxide ("TIPAQUE CR-90," manufactured by ISHIHARA SANGYO KAISHA, LTD.) was added in an amount of 10 parts by weight, to give Powder Coating 10-8. The

triboelectric charge of Powder Coating 10-8 was -15.7  $\mu\text{C/g}$ , the apparent density was 0.415 g/cc, and the softening point was 114°C.

5     Example J-1

25 parts by weight of Powder Coating 10-1, 37.5 parts by weight of Powder Coating 10-2, and 37.5 parts by weight of Powder Coating 10-3 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate using an electrostatic sprayer, and the coated steel substrate was stoved at 180°C for 20 minutes to form a coating film. The formed coating film was visually homogeneous red. The maximum differences in the triboelectric charges, in the apparent densities, and in the softening points of each the powder coatings blended are respectively shown in Table 10.

Example J-2

25 parts by weight of Powder Coating 10-1, 37.5 parts by weight of Powder Coating 10-3, and 37.5 parts by weight of Powder Coating 10-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example J-1 to form a coating film. The formed coating film was visually homogeneous green. The maximum differences in the

triboelectric charges, in the apparent densities, and in the softening points of each the powder coatings blended are respectively shown in Table 10.

5     Example J-3

25 parts by weight of Powder Coating 10-1, 37.5 parts by weight of Powder Coating 10-2, and 37.5 parts by weight of Powder Coating 10-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example J-1 to form a coating film. The formed coating film was visually homogeneous blue. The maximum differences in the triboelectric charges, in the apparent densities, and in the softening points of each the powder coatings blended are respectively shown in Table 10.

Example J-4

25 parts by weight of Powder Coating 10-1, 56.3 parts by weight of Powder Coating 10-3, and 18.7 parts by weight of Powder Coating 10-4 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example J-1 to form a coating film. The formed coating film was visually homogeneous yellow green. The maximum differences in the triboelectric charges, in the apparent densities, and in

the softening points of each the powder coatings blended are respectively shown in Table 10.

Example J-5

5           25 parts by weight of Powder Coating 10-1, 18.7 parts  
by weight of Powder Coating 10-3, and 56.3 parts by weight  
of Powder Coating 10-4 were blended using a Henschel  
mixer. The resulting mixture was applied to a degreased  
steel substrate in the same manner as in Example J-1 to  
10 form a coating film. The formed coating film was visually  
homogeneous blue green. The maximum differences in the  
triboelectric charges, in the apparent densities, and in  
the softening points of each the powder coatings blended  
are respectively shown in Table 10.

15

Example J-6

23 parts by weight of Powder Coating 10-1, 37.5 parts  
by weight of Powder Coating 10-2, 37.5 parts by weight of  
Powder Coating 10-3, and 2 parts by weight of Powder  
20 Coating 10-5 were blended using a Henschel mixer. The  
resulting mixture was applied to a degreased steel  
substrate in the same manner as in Example J-1 to form a  
coating film. The formed coating film was visually  
homogeneous blurred red. The maximum differences in the  
25 triboelectric charges, in the apparent densities, and in

the softening points of each the powder coatings blended are respectively shown in Table 10.

Example J-7

5           15 parts by weight of Powder Coating 10-1, 37.5 parts  
by weight of Powder Coating 10-2, 37.5 parts by weight of  
Powder Coating 10-3, and 10 parts by weight of Powder  
Coating 10-5 were blended using a Henschel mixer. The  
10           resulting mixture was applied to a degreased steel  
substrate in the same manner as in Example J-1 to form a  
coating film. The formed coating film was visually  
homogeneous dark red. The maximum differences in the  
triboelectric charges, in the apparent densities, and in  
the softening points of each the powder coatings blended  
15           are respectively shown in Table 10.

Comparative Example j-1

50 parts by weight of Powder Coating 10-6 and  
50 parts by weight of Powder Coating 10-7 were blended  
20           using a Henschel mixer. The resulting mixture was applied  
to a degreased steel substrate in the same manner as in  
Example J-1 to form a coating film. The formed coating  
film was visually homogeneous blurred red. The maximum  
differences in the triboelectric charges, in the apparent  
25           densities, and in the softening points of each the powder

coatings blended are respectively shown in Table 11.

Comparative Example j-2

50 parts by weight of Powder Coating 10-7 and  
5 50 parts by weight of Powder Coating 10-8 were blended  
using a Henschel mixer. The resulting mixture was applied  
to a degreased steel substrate in the same manner as in  
Example J-1 to form a coating film. The formed coating  
film was visually homogeneous, blurred green. The maximum  
10 differences in the triboelectric charges, in the apparent  
densities, and in the softening points of each the powder  
coatings blended are respectively shown in Table 11.

Comparative Example j-3

15 50 parts by weight of Powder Coating 10-6 and  
50 parts by weight of Powder Coating 10-8 were blended  
using a Henschel mixer. The resulting mixture was applied  
to a degreased steel substrate in the same manner as in  
Example J-1 to form a coating film. The formed coating  
20 film was visually homogeneous, blurred blue. The maximum  
differences in the triboelectric charges, in the apparent  
densities, and in the softening points of each the powder  
coatings blended are respectively shown in Table 11.

25 Comparative Example j-4

75 parts by weight of Powder Coating 10-7 and  
25 parts by weight of Powder Coating 10-8 were blended  
using a Henschel mixer. The resulting mixture was applied  
to a degreased steel substrate in the same manner as in  
5 Example J-1 to form a coating film. The formed coating  
film was visually homogeneous, blurred yellow green. The  
maximum differences in the triboelectric charges, in the  
apparent densities, and in the softening points of each  
the powder coatings blended are respectively shown in  
10 Table 11.

Comparative Example j-5

25 parts by weight of Powder Coating 10-7 and  
75 parts by weight of Powder Coating 10-8 were blended  
15 using a Henschel mixer. The resulting mixture was applied  
to a degreased steel substrate in the same manner as in  
Example J-1 to form a coating film. The formed coating  
film was visually homogeneous blurred blue green. The  
maximum differences in the triboelectric charges, in the  
20 apparent densities, and in the softening points of each  
the powder coatings blended are respectively shown in  
Table 11.

Comparative Example j-6

25 12.5 parts by weight of Powder Coating 10-1,

37.5 parts by weight of Powder Coating 10-2, and 50 parts by weight of Powder Coating 10-7 were blended using a Henschel mixer. The resulting mixture was applied to a degreased steel substrate in the same manner as in Example J-1 to form a coating film. The formed coating film was visually homogeneous red. The maximum differences in the triboelectric charges, in the apparent densities, and in the softening points of each the powder coatings blended are respectively shown in Table 11.



Table 10

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu\text{C}/\text{g}$ ) | Difference in<br>Apparent<br>Densities<br>( $\text{g}/\text{cc}$ ) | Difference in<br>Softening<br>Points<br>( $^{\circ}\text{C}$ ) |
|-------------|--|---|--|--|
| Example J-1 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-2)<br>Powder<br>Coating (10-3) | 1.9   | 0.005  | 2.0  |
| Example J-2 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-3)<br>Powder<br>Coating (10-4) | 1.3   | 0.005  | 2.0  |
| Example J-3 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-2)<br>Powder<br>Coating (10-4) | 1.2   | 0.004  | 1.0  |
| Example J-4 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-3)<br>Powder<br>Coating (10-4) | 1.3   | 0.005  | 2.0  |
| Example J-5 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-3)<br>Powder<br>Coating (10-4) | 1.3   | 0.005  | 2.0  |

Table 10 (continued)

|             |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu\text{C}/\text{g}$ ) | Difference in<br>Apparent<br>Densities<br>( $\text{g}/\text{cc}$ ) | Difference in<br>Softening<br>Points<br>( $^{\circ}\text{C}$ ) |
|-------------|--|---|--|--|
| Example J-6 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-2)<br>Powder<br>Coating (10-3)<br>Powder<br>Coating (10-5) | 2.7   | 0.005  | 3.0  |
| Example J-7 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-2)<br>Powder<br>Coating (10-3)<br>Powder<br>Coating (10-5) | 2.7   | 0.005  | 3.0  |

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Table 11

|                            |  | Difference in<br>Triboelectric<br>Charges<br>( $\mu\text{C}/\text{g}$ ) | Difference in<br>Apparent<br>Densities<br>( $\text{g}/\text{cc}$ ) | Difference in<br>Softening<br>Points<br>( $^{\circ}\text{C}$ ) |
|----------------------------|--|---|--|--|
| Comparative<br>Example j-1 | Powder<br>Coating (10-6)<br>Powder<br>Coating (10-7)                             | 2.3   | 0.001  | 1.0  |
| Comparative<br>Example j-2 | Powder<br>Coating (10-7)<br>Powder<br>Coating (10-8)                             | 0.4   | 0.001  | 1.0  |
| Comparative<br>Example j-3 | Powder<br>Coating (10-6)<br>Powder<br>Coating (10-8)                             | 1.9   | 0.000  | 0.0  |
| Comparative<br>Example j-4 | Powder<br>Coating (10-7)<br>Powder<br>Coating (10-8)                             | 0.4   | 0.001  | 1.0  |
| Comparative<br>Example j-5 | Powder<br>Coating (10-7)<br>Powder<br>Coating (10-8)                             | 0.4   | 0.001  | 1.0  |
| Comparative<br>Example j-6 | Powder<br>Coating (10-1)<br>Powder<br>Coating (10-2)<br>Powder<br>Coating (10-7) | 0.8   | 0.004  | 3.0  |

Test Example

With respect to the coating films obtained in each of Examples J-1 to J-7 and Comparative Examples j-1 to j-6, the hue of the coating films was measured using a spectrodensitometer "X RITE" (manufactured by X Rite, Inc.). Incidentally, in Table 12, L stands for brightness. Higher the  $a^*$  values, stronger the red in the hue of the coating films, and lower the  $a^*$  values, stronger the green in the hue of the coating films. Similarly, higher the  $b^*$  values, stonger the yellow in the hue of the coating films, and lower the  $b^*$  values, stronger the blue in the hue of the coating films. Also,  $c^*$  stands for chroma of the hue, wherein  $c^* = [(a^*)^2 + (b^*)^2]^{0.5}$ .

15

T a b l e 1 2

|                            | Brightness | Hue   |       | Chroma |
|----------------------------|------------|-------|-------|--------|
|                            | L          | a *   | b *   | c *    |
| Example J-1                | 38.9       | 50.6  | 26.3  | 57.0   |
| Example J-2                | 39.0       | -35.2 | 11.6  | 37.1   |
| Example J-3                | 21.2       | 13.7  | -31.8 | 34.6   |
| Example J-4                | 45.6       | -37.3 | 29.9  | 47.8   |
| Example J-5                | 33.2       | -25.4 | -11.0 | 27.7   |
| Example J-6                | 31.0       | 40.4  | 22.1  | 46.0   |
| Example J-7                | 19.5       | 13.3  | 4.6   | 14.1   |
| Comparative<br>Example j-1 | 31.2       | 38.9  | 19.8  | 43.6   |
| Comparative<br>Example j-2 | 37.1       | -21.5 | 10.3  | 23.8   |
| Comparative<br>Example j-3 | 19.3       | 10.7  | -20.3 | 22.9   |
| Comparative<br>Example j-4 | 39.7       | -26.7 | 28.7  | 39.2   |
| Comparative<br>Example j-5 | 35.2       | -10.5 | -11.0 | 15.2   |
| Comparative<br>Example j-6 | 33.1       | 39.6  | 22.9  | 45.7   |

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As a result, as shown in Table 12, the range of values for Examples J-1 to J-7 and Comparative Examples j-1 to j-6 are as follows.

L values:

5           Examples: 19.5 to 45.6.

          Comparative Examples: 19.3 to 39.7.

a\* values:

          Examples: -37.3 to +50.6.

          Comparative Examples: -26.7 to +39.6.

10          b\* values:

          Examples: -31.8 to +29.9.

          Comparative Examples: -20.3 to +28.7.

c\* values:

          Examples: 14.1 to 57.0.

15          Comparative Examples: 15.2 to 45.7.

These values for brightness, hue and chroma are in notably wide ranges for Examples in the present invention.

The present invention being thus described, it will  
20   be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention, and all such modifications as would be obvious to one skilled in the art are intended to be included within the scope of the  
25   following claims.

WHAT IS CLAIMED IS:

1. A combination of powder coatings usable in a powder coating method for forming a coating film having  
5 visually homogeneous hue by mixing two or more powder coatings of which each color is different, wherein each of differences in triboelectric charges of said two or more powder coatings is 5.0  $\mu\text{C/g}$  or less.

10 2. The combination of powder coatings according to claim 1, wherein each of differences in true specific gravities of said two or more powder coatings is 0.15 g/cc or less.

15 3. The combination of powder coatings according to claim 1, wherein each of differences in apparent densities of said two or more powder coatings is 0.020 g/cc or less.

20 4. The combination of powder coatings according to claim 1, wherein each of differences in softening points of said two or more powder coatings is 5.0°C or less, the softening points being measured using a capillary rheometer.

25 5. The combination of powder coatings according to

claim 1, wherein each of differences in dielectric constants of said two or more powder coatings is 0.20 or less.

5           6.    The combination of powder coatings according to claim 1, wherein each of ratios of the electric resistance of said two or more powder coatings is from 0.1 to 10.

10           7.    The combination of powder coatings according to claim 1, wherein at least one powder coating is a white powder coating containing a white pigment, and the remaining powder coatings comprise no white pigments.

15           8.    A powder coating composition usable in a powder coating method for forming a coating film having visually homogeneous hue, wherein said powder coating composition comprises two or more powder coatings selected from the combination of any one of claims 1 to 7.

20           9.    A coating method for forming a coating film having visually homogeneous hue, comprising the step of applying onto a substrate to be coated two or more powder coatings of which each color is different, wherein each of differences in triboelectric charges of said two or more  
25   powder coatings is 5.0  $\mu\text{C/g}$  or less.



5            11. The coating method according to claim 9, wherein  
each of differences in apparent densities of said two or  
more powder coatings is 0.020 g/cc or less.

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coatings comprise no white pigments.

16. A coating method for forming a coating film having visually homogeneous hue, comprising the steps of  
5 applying a white coating on a substrate to be coated, to give a substrate coated with the white coating; and then applying thereon two or more powder coatings of which each color is different, wherein each of differences in  
10 triboelectric charges of said two or more powder coatings is 5.0  $\mu\text{C/g}$  or less.

17. The coating method according to claim 16,  
wherein each of differences in true specific gravities of  
15 said two or more powder coatings is 0.15 g/cc or less.

18. The coating method according to claim 16,  
wherein each of differences in apparent densities of said  
two or more powder coatings is 0.020 g/cc or less.

19. The coating method according to claim 16,  
20 wherein each of differences in softening points of said two or more powder coatings is 5.0°C or less, the softening points being measured using a capillary  
rheometer.

25

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21. The coating method according to claim 16,  
wherein each of ratios of the electric resistance of said  
two or more powder coatings is from 0.1 to 10.



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## ABSTRACT OF THE DISCLOSURE

A combination of powder coatings usable in a powder coating method for forming a coating film having visually  
5 homogeneous hue by mixing two or more powder coatings of which each color is different. In the combination of powder coatings, each of differences in triboelectric charges of two or more powder coatings is 5.0  $\mu\text{C/g}$  or less. The coating method usable herein includes the step  
10 of applying onto a substrate to be coated two or more powder coatings of which each color is different, in which the triboelectric charges are controlled in the above given ranges.

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# BIRCH, STEWART, KOLASCH & BIRCH, LLP

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ATTORNEY DOCKET NO.

### FOR PATENT AND DESIGN APPLICATIONS

1422-297P

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As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated next to my name; that I verily believe that I am the original, first and sole inventor (if only one inventor is named below) or an original, first and joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:\*

Insert Title

POWDER COATING

Check Box If  
Appropriate -  
For Use Without  
Specification  
Attached

the specification of which is attached hereto unless the following box is checked:

☐ was filed on \_\_\_\_\_ as United  
States Application Number \_\_\_\_\_ or  
PCT International Application Number \_\_\_\_\_  
and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I do not know and do not believe the same was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof, or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months (six months for designs) prior to this application, and that no application for patent or inventor's certificate on this invention has been filed in any country foreign to the United States of America prior to this application by me or my legal representatives or assigns, except as follows.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

#### Prior Foreign Application(s)

Priority Claimed

Insert Priority  
Information  
(if appropriate)

|                      |                    |  |   |                             |
|----------------------|--------------------|--|---|-----------------------------|
| 8-84846<br>(Number)  | Japan<br>(Country) | March 12, 1996<br>(Month/Day/Year Filed) | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| 8-106242<br>(Number) | Japan<br>(Country) | April 2, 1996<br>(Month/Day/Year Filed)  | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| 8-113223<br>(Number) | Japan<br>(Country) | April 9, 1996<br>(Month/Day/Year Filed)  | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| 8-135753<br>(Number) | Japan<br>(Country) | May 1, 1996<br>(Month/Day/Year Filed)    | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |
| 8-171763<br>(Number) | Japan<br>(Country) | June 10, 1996<br>(Month/Day/Year Filed)  | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No |

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

All Foreign Applications, if any, for any Patent or Inventor's Certificate Filed More Than 12 Months (6 Months for Designs) Prior To The Filing Date of This Application:

Country

Application No.

Date of Filing (Month/Day/Year)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Number)

(Filing Date)

(Status — patented, pending, abandoned)

(Application Number)

(Filing Date)

(Status — patented, pending, abandoned)

\*NOTE: Must be completed.

(\* Continued)

| Insert Priority<br>Information<br>(if appropriate) | Prior Foreign Application(s) |                    |   | Priority                                   | Claimed                        |
|--|------------------------------|--------------------|---|--|--------------------------------|
| ■  | 8-188202<br>(Number)         | Japan<br>(Country) | June 28, 1996<br>(Month/Day/Year Filed) | <input checked="" type="checkbox"/><br>Yes | <input type="checkbox"/><br>No |
|  | _____<br>(Number)            | _____<br>(Country) | _____<br>(Month/Day/Year Filed)         | <input type="checkbox"/><br>Yes            | <input type="checkbox"/><br>No |
|  | _____<br>(Number)            | _____<br>(Country) | _____<br>(Month/Day/Year Filed)         | <input type="checkbox"/><br>Yes            | <input type="checkbox"/><br>No |
|  | _____<br>(Number)            | _____<br>(Country) | _____<br>(Month/Day/Year Filed)         | <input type="checkbox"/><br>Yes            | <input type="checkbox"/><br>No |
|  | _____<br>(Number)            | _____<br>(Country) | _____<br>(Month/Day/Year Filed)         | <input type="checkbox"/><br>Yes            | <input type="checkbox"/><br>No |

462120 25557800

I hereby appoint the following attorneys to prosecute this application and/or an international application based on this application and to transact all business in the Patent and Trademark Office connected therewith and in connection with the resulting patent based on instructions received from the entity who first sent the application papers to the attorneys identified below, unless the inventor(s) or assignee provides said attorneys with a written notice to the contrary:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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I hereby appoint the following attorneys to prosecute this application and/or an international application based on this application and to transact all business in the Patent and Trademark Office connected therewith and in connection with the resulting patent based on instructions received from the entity who first sent the application papers to the attorneys identified below, unless the inventor(s) or assignee provides said attorneys with a written notice to the contrary:

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Inventor, if any:

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